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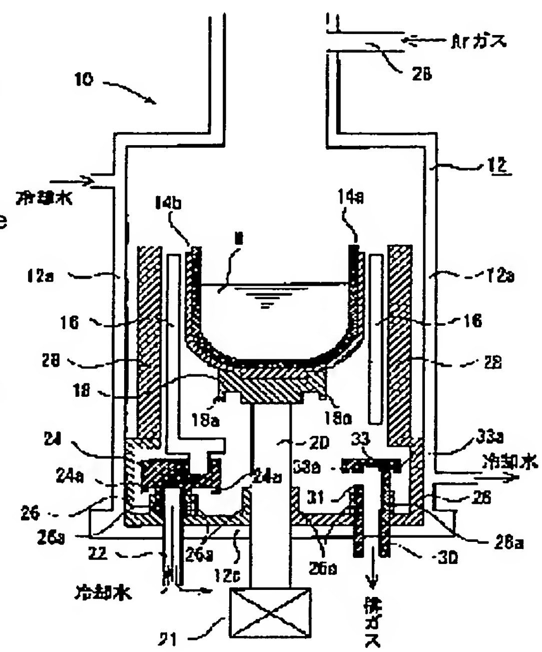
(54) SEMICONDUCTOR SINGLE CRYSTAL PRODUCTION DEVICE AND METHOD OF EVALUATING GRAPHITE MEMBER

(57)Abstract:
PROBLEM TO BE SOLVED: To prevent an outflow of a high temperature raw material to the outside of a production device and corrosion of the wall of a device furnace by the flown-out raw material in the case when the high temperature raw material is flown out by an accident in a semiconductor single crystal production device based on a CZ method, and to provide a method of evaluating a graphite member suitable for preventing the outflow.
SOLUTION: A quartz crucible 14a is provided at the inside of a furnace 12 of a production device. A protective crucible 14b, a crucible stand 18 and a crucible supporting shaft 20, which may be brought into contact with a molten raw material M when it flows out, are constituted of graphite members. Isotropic graphite having a bulk density of 1.70 to 1.90 g/cm3 is used for producing the graphite members. Or, a graphite material having such a characteristic that the rate of dimension change of a part dipped in a silicon melt for a time between 10 to 20 min is ≤5% is used for producing the graphite members. Further, isotropic graphite such that its ash content is ≤20 ppm is used.



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CLAIMS

[Claim(s)]
[Claim 1] In the manufacturing installation of a semi-conductor single crystal which raises a single crystal with the Czochrlski method which has the heating heater for carrying out heating fusion of the raw material melt and raw material melt which were held by the crucible in the furnace of a manufacturing installation, protection of a manufacturing installation furnace wall, and the graphite member arranged for the incubation in a furnace etc. The graphite member arranged in the manufacturing installation furnace which may touch the raw material melt which flowed out when a melting raw material flowed out of the crucible which held raw material melt at least, Or the manufacturing installation of the semi-conductor single crystal characterized by bulk density using the isotropic graphite of 1.70 - 1.90 g/cm3 for the graphite member arranged in the manufacturing installation furnace for preventing holding the raw material melt which flowed out and contacting a manufacturing installation furnace wall as an ingredient of this graphite member.
[Claim 2] In the manufacturing installation of a semi-conductor single crystal which raises a single crystal with the Czochrlski method which has the heating heater for carrying out heating fusion of the raw material melt and raw material melt which were held by the crucible in the furnace of a manufacturing installation, protection of a manufacturing installation furnace wall, and the graphite member arranged for the incubation in a furnace etc. The graphite member arranged in the manufacturing installation furnace which may touch the raw material melt which flowed out when a melting raw material flowed out of the crucible which held raw material melt at least, or as an ingredient of the graphite member arranged in a manufacturing installation furnace in order to prevent holding the raw material melt which flowed out and contacting a manufacturing installation furnace wall The manufacturing installation of the semi-conductor single crystal characterized by using the graphite material whose rate of a dimensional change of the part which was immersed in the fused silicon melt less than 20 minutes 10 minutes or more, and was immersed in subsequent silicon melt is 5% or less.
[Claim 3] the graphite member which may touch outflow melt in the graphite member which is the manufacturing installation of the semi-conductor single crystal indicated to claim 1 or claim 2, and has been arranged in the furnace of said semi-conductor single crystal manufacturing installation at least when raw material melt flows out of a crucible -- a graphite crucible, a crucible base, a crucible support shaft, the graphite protection polar zone, a heating heater, and an exhaust gas pipe -- and -- or the manufacturing installation of the semi-conductor single crystal characterized by being a molten-bath leakage saucer.
[Claim 4] The manufacturing installation of the semi-conductor single crystal which is the manufacturing installation of the semi-conductor single crystal indicated in any 1 term of claim 1 - claim 3, and is characterized by ash content using an isotropic graphite 20 ppm or less as an ingredient of said graphite member.
[Claim 5] The evaluation approach of the graphite material for semi-conductor single crystal manufacturing installations which carries out the description of evaluating the property of this graphite material from the dimension or volume change of the graphite ingredient part which was immersed in the raw material melt of the obtained semi-conductor single crystal which carried out heating fusion in graphite material, and was immersed in raw material melt.
[Claim 6] Said raw material melt immersed in the graphite material which is the graphite material evaluation approach for semiconductor devices indicated to claim 5, and serves as a candidate for evaluation is the evaluation approach of the graphite material for semiconductor devices characterized by being silicon melt which carried out heating fusion and obtained polycrystalline silicon or single crystal silicon.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[The technical field to which invention belongs] This invention relates to the evaluation approach of the graphite components ingredient used for the manufacturing installation and manufacturing installation of the semi-conductor single crystal for manufacturing a semi-conductor single crystal. It is the Czochrlski method (Czochralski Method and a following CZ process are called.) which grows a single crystal under the seed crystal by being immersed and pulling up a crystal to the raw material melt held in the crucible in a single crystal manufacturing installation furnace in more detail. It is related with the graphite material evaluation approach of evaluating the property of the graphite component which constitutes the manufacturing installation of the used semi-conductor single crystal, and its graphite component.
[0002]
[Description of the Prior Art] The CZ process which dissolves a semi-conductor single crystal raw material within the crucible of semiconductor fabrication machines and equipment, is made to **** seed crystal to this melt side as one of the manufacture approaches of a semi-conductor single crystal conventionally, and raises a semi-conductor single crystal is known widely. Although the training technique of the semi-conductor single crystal using a CZ process is broadly used in order to obtain semi-conductor single crystals, such as silicon and GaAs, below, it explains the conventional example of this invention by making training of a silicon semi-conductor single crystal into the example.
[0003] After the method of raising a silicon single crystal by the CZ process fills up with the polycrystalline silicon used as a raw material the crucible by which the outside which it had in the manufacturing installation furnace was constituted from a product made from a graphite, and the inside was constituted from a product made from a quartz and fills the inside of a manufacturing installation furnace with inert gas, such as an argon (it is also hereafter called Ar.), it is heated to an elevated temperature 1400 degrees C or more at the heating heater arranged in the perimeter of an outside of a crucible, and dissolves a raw material. And the semi-conductor single crystal of silicon is formed under the seed crystal by pulling up up, rotating seed crystal calmly, if take down seed crystal from the upper part to this silicon melt by which heating melting was carried out, a tip is made to **** to a silicon melt side and the temperature of seed crystal and silicon melt is stabilized.
[0004] In training of such a semi-conductor single crystal, while growing up the single crystal, it is necessary to continue holding within a crucible the melting liquid which is the raw material in the hot condition which can raise a semi-conductor single crystal. Measures with which the raw material melt which detected the melt outflow in a crucible promptly even if the melt of an elevated temperature [crucible] should flowed out in the manufacturing installation furnace, since possibility of inducing a disaster serious when the hot melt out of a crucible begins to leak in a manufacturing installation furnace by the unforeseen accident of during this period, an earthquake, or others was also considered enough, and flowed out of the crucible does not begin to leak outside a manufacturing installation, either are taken.
[0005] For example, when raw material melt flows into JP,11-180794,A out of a crucible, to it, the fall of the unusual melt side within a crucible is detected promptly, and the equipment which tells an operator about melt having leaked from the inside of a crucible is indicated. It is related with the raw material melt which flowed out of the crucible. And to JP,2-225393,A By devising the configuration and configuration of a graphite member or a heat insulator which have been arranged for protection of a manufacturing installation furnace wall, the incubation in a furnace, etc. The raw material melt which flowed out of the crucible is held in a manufacturing installation furnace. Begin to leak to the outside of a manufacturing installation, or Or in order to cool the manufacturing installation furnace wall made with the metal, it

prevents that hot raw material melt eats away the furnace wall of the single crystal manufacturing installation which made the furnace wall dual structure and was carried out [flow back / between furnace walls / cooling water], and the single crystal manufacturing installation equipped with the function for preventing the accident by a phreatic explosion etc. is shown, and it is by carrying out.

[0006] By taking these measures, it leaked out of the manufacturing installation furnace, and an operator is exposed to risk or the raw material melt which flowed out of the crucible has avoided the situation whose production of a single crystal carries out damaging an outbreak of a fire and a manufacturing installation etc., and becomes impossible. As for the hot raw material melt which might cause serious disaster, such as a phreatic explosion, when the furnace wall was eaten away by hot melt, even when it was small, and leaked from the crucible, in the single crystal manufacturing installation which flowed back cooling water to the furnace wall in order to protect a metal manufacturing installation furnace wall from the elevated-temperature ambient atmosphere in a furnace especially, it is important to catch certainly by the graphite member arranged in a manufacturing installation furnace by the above equipment and approaches.

[0007]

[Problem(s) to be Solved by the Invention] However, the configuration and structure of the graphite member which has protected the furnace wall from the crucible made from a graphite which constitutes the interior of a furnace of a single crystal manufacturing installation, or a heating heater or hot radiant heat are made suitable. Even when raw material melt leaks from a crucible, even if it perceives it promptly and stops a single crystal manufacturing installation The radiationnal-cooling time amount which the temperature in a manufacturing installation furnace falls and attains to safe temperature by **** also in 3 - 5 hours is required. Without making the raw material melt which leaked flow out out of a manufacturing installation furnace in the meantime, or making a furnace wall touched, certainly, it must hold with the member and heat insulator made from a graphite in a manufacturing installation furnace, and must place.

[0008] Especially, in order to raise the semi-conductor single crystal of a large diameter in manufacture of the latest silicon semi-conductor single crystal, the heat capacity of equipment itself is also large, and by the time it stops the electric power supply to a heating heater, the temperature inside equipment falls and it can open safely, it is necessary for installation of the large-sized single crystal manufacturing installation which can hold the silicon melt of 100kg or more thing large capacity to also progress, and to perform still longer time amount radiationnal cooling by such large-sized single crystal manufacturing installation. It becomes important to make the member which needs to hold certainly, without leaking to the exterior the melt which flowed out with the graphite member which constitutes the interior of a furnace, or the heat insulator made from a graphite until whenever [manufacturing installation furnace temperature] reaches safe temperature, even if it is the case where raw material melt flows out of a crucible under such a situation with a natural thing, chooses a more reliable ingredient in order to hold the melt which leaked certainly, and touches outflow melt.

[0009] Moreover, although it is in operation in training of a semi-conductor single crystal by filling the inside of a manufacturing installation furnace with reactant inert gas, such as low Ar, for protection of the components which constitute the inside of maintenance and the manufacturing installation furnace of single crystal quality From the raw material melt heated by the elevated temperature at the heater, matter, such as SiO (oxide of silicon) with fluxing action, has always evaporated. The graphite member and the heat insulator made from a graphite which have been arranged by these emissions in a manufacturing installation furnace may receive pervasion, and it may lose by pervasion while operating reinforcement and an incubation function depending on the class of graphite ingredient which forms each part material. The graphite member in the manufacturing installation furnace at which the outflow melt from a crucible touches is asked also for demonstrating the function stabilized without spoiling the function when a long duration pan is carried out to the emission which evaporated from raw material melt.

[0010] In the semi-conductor single crystal manufacturing installation in view of such a problem, accomplish this invention, and according to a CZ process When hot raw material melt flows out of the crucible put on the interior of a single crystal manufacturing installation furnace according to an unexpected cause The manufacturing installation of the semi-conductor single crystal equipped with the graphite member arranged in the manufacturing installation furnace which has the function for hot raw material melt not to corrode the furnace wall of a semi-conductor single crystal manufacturing installation, either, but to hold raw material melt safely further, without making an outflow raw material flow out out of a stop manufacturing installation certainly in a furnace, It aims at offering the ingredient evaluation approach of the graphite member which constitutes the semi-conductor single crystal manufacturing installation which can choose appropriately the graphite material used as the ingredient of a graphite member suitable for it.

[0011]
[Means for Solving the Problem] In order to solve the above-mentioned technical problem, the 1st mode of the semi-conductor single crystal manufacturing installation of this invention In the manufacturing installation of a semi-conductor single crystal which raises a single crystal with the Czochrlski method which has the heating heater for carrying out heating fusion of the raw material melt and raw material melt which were held by the crucible in the furnace of a manufacturing installation, protection of a manufacturing installation furnace wall, and the graphite member arranged for the incubation in a furnace etc. To the graphite member arranged in the manufacturing installation furnace for protecting, holding the graphite member or the raw material melt which flowed out arranged in the manufacturing installation furnace which may touch the raw material melt which flowed out when a melting raw material flowed out of the crucible which held raw material melt at least, and contacting a manufacturing installation furnace wall Bulk density is characterized by using the isotropic graphite of 1.70 - 1.90 g/cm3 as an ingredient of this graphite member.
[0012] Thus, the graphite member which is in the location which touches outflow melt when raw material melt leaks from a crucible It is cold isostatic pressing (Cold Isostatic Press and Following CIP are called.) as an ingredient of a graphite member which it is desirable to choose and arrange the ingredient which cannot receive pervasion of raw material melt easily, and fulfills this condition. It is most desirable to use the isotropic graphite by which shaping manufacture was used and carried out and in which bulk density has the property of 1.70 - 1.90 g/cm3.
[0013] Since raw material melt permeates the interior of a graphite member quickly and a rapid cubical expansion arises when the graphite member to which bulk density touches raw material melt using three or less 1.70 g/cm graphite material is created, it cannot become impossible to maintain the function as the structure, raw material melt cannot be stopped in a furnace, but the metal sections, such as a manufacturing installation furnace wall, may be made to eat away, or the melt which leaked out of the equipment furnace may be made to flow out. Moreover, even if metaphor bulk density is three or more 1.70 g/cm, in extrusion molding (Extruding Press) and the anisotropy graphites which carried out die pressing and were made using shaping (Molding Press) etc. other than an isotropic graphite, there is a problem in respect of endurance or dependability that it is easy to receive pervasion of the emission which comes out of raw material melt at the time of single crystal growth. Therefore, it is suitable for the graphite components which may touch an outflow raw material when raw material melt leaks from a crucible that bulk density uses a three or more 1.70 g/cm isotropic graphite. However, since, as for the isotropic graphite material with the value of bulk density high beyond the need, a manufacturing cost also increases on the other hand and it will become very expensive, stopping to about three 1.90 g/cm is appropriate for the upper limit of bulk density.
[0014] And in the graphite member arranged in the furnace of a semi-conductor single crystal at least The graphite crucible which may touch outflow melt when raw material melt flows out of a crucible, If the bulk density which has the above-mentioned property makes a crucible base, a crucible support shaft, the graphite protection polar zone, a heating heater, an exhaust gas pipe, and a molten-bath leakage saucer from the isotropic graphite of 1.70 - 1.90 g/cm3 Since neither deformation nor a cracking crack is produced in a member even if raw material melt leaks from a crucible and it touches the graphite member in a manufacturing installation furnace, the raw material melt which flowed out certainly by these graphites member can be held.
[0015] Moreover, in the single crystal manufacturing installation of a silicon semi-conductor, if bulk density makes and arranges the graphite member in a furnace with the isotropic graphite of 1.70 - 1.90 g/cm3, SiO (oxide of silicon) which evaporates from raw material melt at the time of single crystal growth permeates the interior of a graphite member gradually, and expansion of the cracking crack and member which are produced by SiC-izing, or a strong fall can be controlled. While the endurance of a graphite member can improve by this and it can be equal now to prolonged use, it becomes possible for the dependability of the graphite components itself to also increase and to take the cure of more positive melt leakage.
[0016] The 2nd mode of the semi-conductor single crystal manufacturing installation which manufactures a semi-conductor single crystal by the CZ process of this invention In the manufacturing installation of a semi-conductor single crystal which raises a single crystal with the Czochrlski method which has the heating heater for carrying out heating fusion of the raw material melt and raw material melt which were held by the crucible in the furnace of a manufacturing installation, protection of a manufacturing installation furnace wall, and the graphite member arranged for the incubation in a furnace etc. As an ingredient of the graphite member arranged in a manufacturing installation furnace in order to prevent holding the graphite member or the raw material melt which flowed out arranged in the manufacturing installation furnace which may touch

the raw material melt which flowed out when a melting raw material flowed out of the crucible which held raw material melt at least, and contacting a manufacturing installation furnace wall The rate of a dimensional change of the part which was immersed in the fused silicon melt less than 20 minutes 10 minutes or more, and was immersed in subsequent silicon melt is characterized by using the graphite material which is 5% or less.

[0017] When graphite material is sunk into the raw material melt same as an ingredient of the graphite member which may touch the hot raw material melt which leaked from the crucible arranged in the furnace of a single crystal manufacturing installation as the single crystal to raise, even if the rate of change of the dimension of the immersion part uses 5% or less of graphite ingredient, the semi-conductor single crystal manufacturing installation of the 1st mode of this invention and a single crystal manufacturing installation with the same description can be obtained.

[0018] If the graphite member to which the rate of a dimensional change at the time of carrying out fixed time amount immersion of the graphite material may touch hot outflow melt at raw material melt using less than 5% of graphite material is made, the property as a graphite member can be maintained without causing big deformation, a cracking crack and expansion, or a fall on the strength, when raw material melt is touched. Moreover, since it is hard to be eaten away also from raw material melt, such as SiO generated at the time of single crystal growth, to an emission, if it can be equal also to the use covering a long time enough and a single crystal manufacturing installation is made using the graphite member using such an ingredient, without the endurance of a member spoiling a function highly, even when raw material melt leaks from a crucible according to an unexpected cause, it can consider as a manufacturing installation with the high dependability which can suppress damage to the minimum.

[0019] And when the melt immersed in graphite material is silicon, time amount immersed in melt in a test member should be made 10 minutes or more. As for the time amount which the big difference to the result from which it is acquired even if the time amount immersed in silicon melt cannot know the right property of an ingredient and is immersed for a long time beyond the need in 10 or less minutes, since silicon melt does not fully permeate the interior of an ingredient is not seen, and is immersed in melt in the test member for the slack reason, it is efficient to stop that it is long in about 20 or less minutes.

[0020] And in the graphite member arranged in the furnace of a semi-conductor single crystal at least The graphite crucible which may touch outflow melt when raw material melt flows out of a crucible, A crucible base, a crucible support shaft, the graphite protection polar zone, a heating heater, an exhaust gas pipe, and a molten-bath leakage saucer If the rate of a dimensional change of the part which was immersed in the fused silicon melt which has the above-mentioned property less than 20 minutes 10 minutes or more, and was immersed in subsequent silicon melt makes from the graphite material which is 5% or less Since neither deformation nor a cracking crack is produced in a member even if raw material melt leaks from a crucible and it touches the graphite member in a manufacturing installation furnace, the raw material melt which flowed out certainly by these graphites member can be held.

[0021] Moreover, for the purity of the graphite member used for the manufacturing installation of a semi-conductor single crystal, these graphites member to which it may be desirable to which that it will be a high grade if the effect of the impurity to training crystal quality is taken into consideration, and it may touch outflow melt is also "JIS. If the ash content when measuring by R 7223" constitutes using the graphite material which is 20 ppm or less, it can consider as the equipment which fitted semi-conductor single crystal manufacture more.

[0022] The evaluation approach of the graphite material for semi-conductor single crystal manufacturing installations by this invention for getting to know the property of the graphite material for single crystal manufacturing installations is characterized by evaluating the property of this graphite material from the dimension or volume change of the graphite ingredient part which was immersed in the raw material melt of the obtained semi-conductor single crystal which carried out heating fusion in graphite material, and was immersed in raw material melt.

[0023] In evaluating the property of the graphite member which constitutes the interior of a manufacturing installation of a semi-conductor single crystal A test sample is immersed in the raw material melt of the single crystal by which started the test sample from the graphite material used as interior material of a furnace, and melting was carried out within the manufacturing installation. the configuration and volume of an immersion part of a test sample are before and after immersion, and if it changed how or a dimension or the volume is measured, evaluating appropriately will cut as a member of which part in a manufacturing installation furnace it is suitable as a member which constitutes the inside of a manufacturing installation furnace, and usable. Since the pervasion degree at the time of raw material adhesion can be exactly known

when evaluating the property of the graphite ingredient which may touch raw material melt especially, it becomes possible to choose the graphite ingredient which has the property searched for more appropriately.
[0024] Moreover, the melt which fused polycrystalline silicon or a silicon single crystal, and was able to do it may be used for the melt immersed in the test sample started from these graphites material. A graphite can obtain an evaluation result by comparatively short time amount, if a reaction progresses quickly, is immersed in silicon melt in the test sample of a graphite and evaluates the property, since the silicon melt by which melting was carried out is easy to be eaten away.
[0025]
[Embodiment of the Invention] Although the example of training of the silicon single crystal using a CZ process is given and explained, referring to an accompanying drawing for the gestalt of operation of this invention below, this invention is not limited only to these. for example, MCZ which raises a semi-conductor single crystal while the evaluation approach of the graphite member used for the manufacturing installation of the semi-conductor single crystal of this invention or its equipment impresses a magnetic field to raw material melt -- naturally it is possible to also use the manufacturing installation of the semi-conductor single crystal using law, and, of course, it also uses for the manufacturing installation of a semi-conductor single crystal which used other CZ processes, such as a compound semiconductor, further -- it is possible.
[0026] Drawing 1 is the outline sectional view showing the gestalt of one operation of the single crystal manufacturing installation of this invention. The manufacturing installation furnace 12 of the single crystal manufacturing installation 10 shown in drawing 1 controls the temperature rise by the hot radiant heat of the heating heater 16 grade which fuses the raw material melt M held in crucible 14made from quartz a, and in order to always consider as constant temperature, it makes dual structure manufacturing installation furnace wall 12a, and it is protected so that a cooling medium may be flowed back and manufacturing installation furnace wall 12a may not become an elevated temperature between the walls of a duplex. In the manufacturing installation 10 of this invention, as a cooling medium, it is flowing back and water is used.
[0027] Moreover, crucible 14made from quartz a which held raw material melt M in the center of the interior of the manufacturing installation furnace 12 is arranged, and since this crucible 14made from quartz a is a product made from a quartz, it is weak against an impact, and since it softens the crucible made from a quartz itself when heated by 1400 more degrees-C or more thing elevated temperature, in order to protect and support it, it has allotted crucible 14made from graphite b made from the graphite outside. And this crucible 14made from graphite b is supported with the crucible support shaft 20 through the crucible base 18, and vertical movement of it is enabled [that rotation is free and] by the crucible rotation drive 21 attached in the lower limit of the crucible support shaft 20.
[0028] Since it is thought that possibility of touching raw material melt M is the highest when the crucible base 18 and the crucible support shaft 20 are also made from graphite material, crucible 12made from graphite b for protecting crucible 12made from quartz a is also combined and hot raw material melt M flows out of crucible 12made from quartz a, the isotropic graphite material of bulk density 1.70 - 1.90 g/cm3 is used.
[0029] Moreover, in order to prevent that the raw material melt M which leaked from crucible 14made from quartz a to the bottom side periphery of the crucible base 18 turns to the crucible support shaft 20, protruding line 18a which becomes a convex toward the bottom is prepared. When raw material melt M is beginning to leak from crucible 14made from quartz a and the crucible base 18 is arrived at, it has prevented preventing leakage melt turning to the crucible support shaft 20 as much as possible, and it being transmitted to the crucible support shaft 20, and beginning to leak out of a furnace by this protruding line 18a.
[0030] On the other hand, immediately, the heating heater 16 made from the graphite for [of graphite crucible 14b] carrying out heating fusion of the raw material melt M outside is placed, and this heating heater 16 is supported by the metal polar zone 22 which supplies the current which heats a heater. When there are protecting from the elevated-temperature ambient atmosphere in the manufacturing installation furnace 12 and an outflow of the raw material melt M from crucible 12made from quartz a, it is joined to the heating heater 16 through the graphite protection polar zone 24 so that pervasion by leakage melt may not be received, and the metal polar zone 22 is performing the electric power supply to the heating heater 16.
[0031] In addition, in order to prevent also heating the metal polar zone 22 beyond the need, cooling water is flowing back like the manufacturing installation furnace wall. Moreover, when raw material melt M is revealed also to the periphery section inferior surface of tongue of the graphite protection polar zone 24 from crucible 14a, protruding line 24a which goes caudad and becomes a convex has surrounded, and the raw material melt M which has carried out the lower stream of a river from the upper part by this protruding

line 24a can be guided to the molten-bath leakage saucer 26 appropriately installed in the lower part of the manufacturing installation furnace 12 so that it may be transmitted to an electrode etc. and may not leak out of a furnace. And since the heating heater 16 and the graphite protection polar zone 24 also have very high possibility that melt **** when raw material melt M leaks from crucible 14made from quartz a, they are using the isotropic graphite material of bulk density 1.70 - 1.90 g/cm3.
[0032] Furthermore, the periphery of the heating heater 16 was equipped with the heat insulator 28 made from a graphite, and it has protected so that the radiant heat from the incubation in the manufacturing installation furnace 12 and heater 16 grade may not hit direct furnace wall 12a.
[0033] And while the molten-bath leakage saucer 26 is arranged at the lower part [of the interior material of a furnace which constitutes the inside of these manufacturing installation furnace 12], and bottom side of the manufacturing installation furnace 12 and protecting **** 12b of the manufacturing installation furnace 12 When raw material melt flows out of crucible 14a according to an unexpected cause The duty holding raw material melt is borne until raw material melt M hot by collecting and catching outflow melt M on this molten-bath leakage saucer 26 eats metal furnace wall 12a away, or it prevents beginning to leak out of the manufacturing installation furnace 12 and the temperature in a furnace falls to a safe temperature. Since it is such, the isotropic graphite material of bulk density 1.70 - 1.90 g/cm3 is used as an ingredient of the molten-bath leakage saucer 26 also with the equipment of this invention.
[0034] This molten-bath leakage saucer 26 needs to protect the interior material 22 of a furnace which bottom wall 12b of the manufacturing installation furnace 12 was protected, and also was prepared in bottom wall 12b, for example, a metal electrode, the polar zone 24 made from a graphite, the crucible support shaft 20, and an exhaust gas pipe 30, and the part of the molten-bath leakage saucer 26 which contacts these members has become annular standup partial 26a so that those parts may be surrounded.
[0035] Moreover, in order to exhaust the gas installation tubing 28 which introduces inert gas in a furnace at a manufacturing installation 10 in order to operate by filling the inside of the manufacturing installation furnace 12 with inert gas in training of a semi-conductor single crystal, and its gas, the exhaust gas pipe 30 is also formed in the lower part in the manufacturing installation furnace 12 so that the molten-bath leakage saucer 26 may be penetrated. With the equipment of this invention, it was made into the structure which discharges the gas in a furnace outside from the exhaust gas pipe 30 in which gas was formed by a sink and the manufacturing installation furnace lower part in the furnace so that Ar gas might be introduced from the gas installation tubing 28 which is above the manufacturing installation furnace and a lower stream of a river might be carried out towards raw material melt at the time of operation.
[0036] Since this exhaust gas pipe 30 may also touch outflow melt when raw material melt M leaks, it is good to protect these by graphite material so that it makes from the isotropic graphite material of bulk density 1.70 - 1.90 g/cm3, or the metal section may be protected like the above-mentioned graphite member in a furnace, when an exhaust gas pipe 30 is a metal. When an exhaust gas pipe 30 is made from the single crystal manufacturing installation 10 of this invention by the isotropic graphite material of the above-mentioned property and the raw material melt M which leaked attains even the molten-bath leakage saucer 26, an exhaust gas pipe 30 is made to set up from the pars basilaris ossis occipitalis of the molten-bath leakage saucer 26, the exhaust gas inlet port 31 is located up so that it may not flow out of the exhaust gas inlet port 31 of an exhaust gas pipe 30, and in order to receive the raw material melt M which falls from the upper part of an exhaust gas pipe 30 further, the upper limit wall 33 is formed in the upper limit of an exhaust gas pipe 30. Protruding line 33a which goes caudad and becomes a convex protrudes, and the melt which fell in the upper limit wall 33 is transmitted in this protruding line 33a, and is brought together in the molten-bath leakage saucer 26 so that leakage melt besides may not leak from the exhaust gas inlet port 31 to the periphery section inferior surface of tongue of an end wall 33 outside.
[0037] In addition, in this invention, the bulk density of graphite material means the mass per unit volume of graphite material (g/cm3). And 5% or less of graphite also has an usable rate of a dimensional change as an ingredient of these graphites member.
[0038] In order to raise a single crystal using the semi-conductor single crystal manufacturing installation of above-mentioned this invention, a polycrystalline silicon raw material is prepared in crucible 14made from quartz a, and after filling the inside of a furnace with inert gas, the raw material melt M which raises a single crystal by melting the polycrystalline silicon raw material which is the lump of silicon at the heating heater 16, and considering as melt is obtained. [0039] Then, a semi-conductor single crystal is raised under the seed crystal by taking down the wire (un-illustrating) for pulling up a single crystal from the upper part, and rolling round a pull-up wire calmly in the place by which temperature was stabilized, after the seed crystal (un-illustrating) attached in the wire lower limit is immersed in raw material melt M.

[0040] Moreover, the erosive evaluation to the raw material melt of the graphite material used as the ingredient of the graphite member of this invention One-side X produces the immersion test sample T of the shape of a rod which die-length Y shows to drawing 2 which is 100mm by 20mm around from the graphite material used as a member. Change to seed crystal and this immersion test sample is attached at the tip of the pull-up wire 35 in the manufacturing installation furnace 12. After making the inside of the equipment furnace 12 into the conditions of the time of single crystal growth, and an abbreviation EQC, the tip of the immersion test sample T made it descend calmly, and was immersed in the test sample T until it sank in raw material melt about 20-30mm. In addition, in drawing 2 , the same as that of drawing 1 or a similar member is shown by the same sign.
[0041] the predetermined time after the test sample T sinks in specified quantity raw material melt -- the test sample T is held in the condition as it is, it pulls up from Melt M after that, the rate of change (deltad) of the dimension of this direction before and after immersing in raw material melt M is measured, and evaluation of erosiveness-proof of the test sample to raw material melt M is performed.
[0042] In addition, the rate of a dimensional change (deltad) is calculated by the following type (1).
[0043]
[Equation 1]

$$\Delta d = \frac{X_2 - X_1}{X_1} \times 100(\%) \quad \cdots \quad (1)$$

[0044] As for X1, in a formula (1), the width of face of the test sample T before being immersed in silicon melt and X2 are the width of face of the test sample T after being immersed in silicon melt.
[0045]
[Example] (Example 1) One side produced [bulk density] the immersion test sample with a die length of 100mm by 20mm around from the isotropic graphite material of 1.74, 1.77, and 1.85 g/cm3.
[0046] It attached at the tip of a pull-up wire of a manufacturing installation as showed these immersion test sample to drawing 2 , and has arranged to the crucible upper part in a manufacturing installation furnace, after filling up the crucible made from a quartz in a manufacturing installation furnace with 10kg polycrystalline silicon after that, the heater was heated and it dissolved, and melt temperature obtained the raw material melt of the silicon around 1420 degrees C.
[0047] It pulls up in the place by which melt temperature was stabilized, and it was being begun to roll a wire, and after taking down the immersion test sample to right above [melt side] and warming it for 30 minutes, the test sample tip was immersed in silicon melt 20mm in the die-length direction. After winding up the stop and the after that pull-up wire for the test sample calmly for 10 minutes and taking out a test sample from melt in silicon melt, appearance observation according sample temperature to change measurement and viewing of the dimension of a return immersion part was gradually performed to ordinary temperature, and the property of graphite material was evaluated.
[0048] the abbreviation as shown to Table 1 and drawing 3 by this evaluation, before the rate of a dimensional change stops at the isotropic graphite material of 1.70 g/cm3 to 4% or less, and neither extreme expansion nor a cracking crack is observed but being immersed in raw material melt -- the same configuration was maintained.
(Example 1 of a comparison) Similarly, with the anisotropy graphite material according [bulk density] to extrusion molding of 1.65 and 1.68 g/cm3, 1.68 g/cm3 carried out die pressing, and bulk density produced the immersion test sample of the same configuration for the example 1 from the anisotropy graphite material by shaping, and performed immersion to silicon melt under the same conditions as an example 1.
[0049] The result was as being shown in Table 1 and drawing 4 , and what the rate of a dimensional change of an immersion part exceeded 5% in all samples, and the cracking crack produced into the immersion part, and the thing from which the configuration changed a lot (expansion) were ******.
[0050]
[Table 1]

（ 黒鉛材のシリコン融液への浸漬評価結果）

実施例 比較例	黒鉛材品種	成形方法	かさ密度 （g／cm³）	融液浸漬後の 寸法変形率	外観観察結果 （ヒビ割れ、変形等）
実施例１	等方性黒鉛	冷間静水圧成形	１．７４	１～２％	亀裂、変形等なし
		冷間静水圧成形	１．７７	１～２％	亀裂、変形等なし
		冷間静水圧成形	１．８５	１％以下	亀裂、変形等なし
比較例１	異方性黒鉛	押出し成形	１．６５	５％以上	浸漬部分が大きく膨張 亀裂が数多く発生
		押出し成形	１．６８	５％以上	浸漬部分が大きく膨張 亀裂が数多く発生
		型押し成形	１．６８	５％以上	浸漬部分が大きく膨張 亀裂が数多く発生

[0051] (Example 2) Next, in the graphite member arranged in a manufacturing installation furnace, when raw material melt leaked from a crucible, bulk density made from 1.74 - 1.77 g/cm3, ash content made the crucible made from a graphite which may touch raw material melt, a crucible base, a crucible support shaft, a heating heater, the graphite protection polar zone, the exhaust gas pipe, and the molten-bath leakage saucer from the isotropic graphite material of a high grade 20 ppm or less, and it was used for manufacture of a silicon semi-conductor single crystal.
[0052] And in raising a semi-conductor single crystal, it turned out that a crack goes into the crucible made from a quartz in the place where about 80% of raw material became with melt about the polycrystalline silicon raw material when 150kg preparation melting was started, and melt is beginning to leak from a crucible at the crucible made from a quartz with an aperture of 60cm. The heating heater was turned off immediately, and when the temperature of the cooling water which is carrying out furnace wall reflux for equipment cooling was checked, especially abnormalities were not seen although water temperature was rising by about 1.2 degrees C.
[0053] Although the silicon around about 80kg had solidified on the molten-bath leakage saucer put on the pars basilaris ossis occipitalis of a manufacturing installation when the manufacturing installation was disassembled and the outflow situation of raw material melt was checked, after the temperature inside a manufacturing installation fell to near the ordinary temperature, there is no trace that the raw material which flowed out leaked from the molten-bath leakage saucer further, and the furnace wall was protected completely. Moreover, when the crucible made from a graphite and crucible base which are considered that the outflow raw material from a crucible touched, and the molten-bath leakage saucer were observed, it turned out that the function as signs that raw material melt received pervasion were not observed but being planned can be demonstrated.
[0054] In addition, this invention is not limited to the above-mentioned gestalt of operation. The above-mentioned gestalt of operation is mere instantiation, and if it has the same configuration substantially with the technical thought indicated by the claim of this invention and the same effectiveness is done so, no matter it may be what thing, being included by the technical range of this invention is undoubted.
[0055] for example, MCZ which raises a semi-conductor single crystal while impressing a magnetic field to raw material melt although the CZ process which raises a single crystal without impressing a magnetic field to raw material melt for the manufacturing installation of the semi-conductor single crystal of this invention was mentioned as the example and explained -- it cannot be overemphasized that the same effectiveness is acquired also in the manufacturing installation of the semi-conductor single crystal using law. And although the property of a graphite member that the method of evaluating the property of the graphite member used for the semi-conductor single crystal manufacturing installation and semi-conductor single crystal manufacturing installation of this invention also constitutes the manufacturing installation of semi-conductor single crystals other than silicon and its equipment is evaluated, naturally it is available, and even when [which used the CZ process] it applies, for example to training of compound semiconductors, such as a GaAs crystal, the effectiveness can fully be demonstrated.
[0056]
[Effect of the Invention] If the manufacturing installation of the semi-conductor single crystal by the CZ process is made into the structure of the equipment of this invention as stated above Since neither big deformation nor a cracking crack is produced in a graphite member even if the raw material melt which has erosiveness in the graphite member which constitutes the inside of a manufacturing installation furnace touches Even if hot raw material melt leaked from the crucible arranged by the metaphor unexpected cause in an equipment furnace, while being able to lead to the graphite member for holding outflow melt, such as a

molten-bath leakage saucer arranged in a furnace in the raw material melt which leaked, correctly After checking the leakage of the raw material melt out of a crucible, long duration until it stops a manufacturing installation and the temperature in a furnace falls to safe temperature is covered, and it becomes possible to stop outflow melt in a furnace certainly by these graphites member.

[0057] Since it is lost that the raw material melt which leaked from the crucible by this flows out out of a manufacturing installation, insurance can be coped with at a trouble, without exposing the operator who is working about [that it becomes avoidable to damage equipment by the hot melt which flowed out], and near the equipment to risk.

[0058] Furthermore, since it becomes without the erosive hot high raw material melt which flowed out of the crucible touching directly the metal manufacturing installation furnace wall which flowed back cooling water, a heater electrode, a crucible driving shaft, etc., serious disaster, such as a phreatic explosion which happens when a hot liquid is mixed with cooling media, such as water, is also avoidable.

[0059] And if the graphite ingredient used as a graphite member in a single crystal manufacturing installation furnace using the evaluation approach of this invention is evaluated, the graphite ingredient suitable for each single crystal manufacturing installation can be known now simply and more certainly, and the dependability of the graphite member arranged in these manufacturing installations will also improve further.

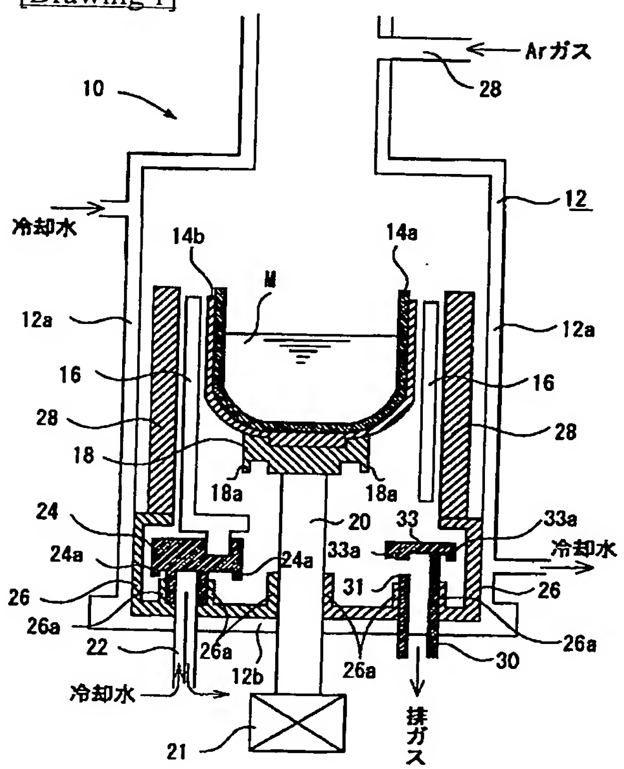
[Translation done.]

* NOTICES *

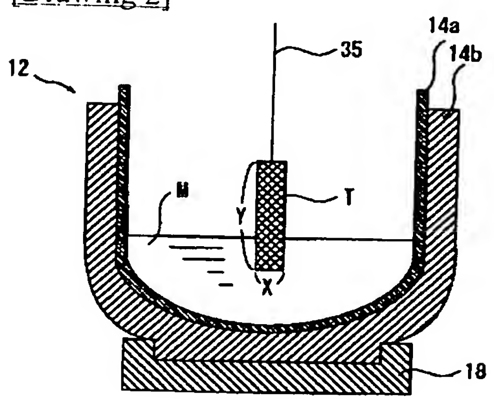
1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DRAWINGS

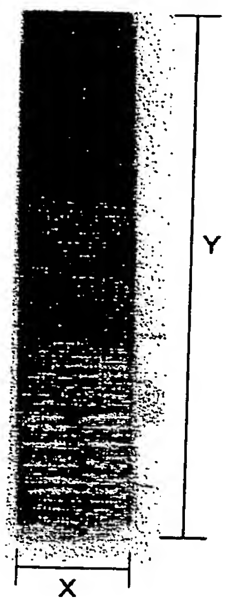
[Drawing 1]



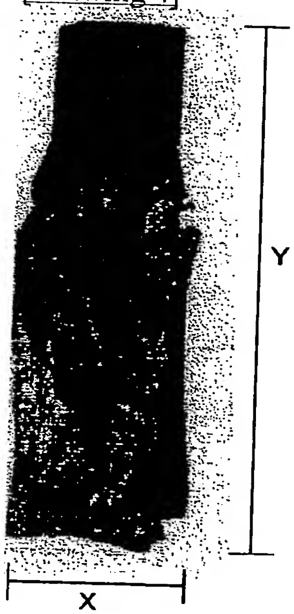
[Drawing 2]



[Drawing 3]



[Drawing 4]



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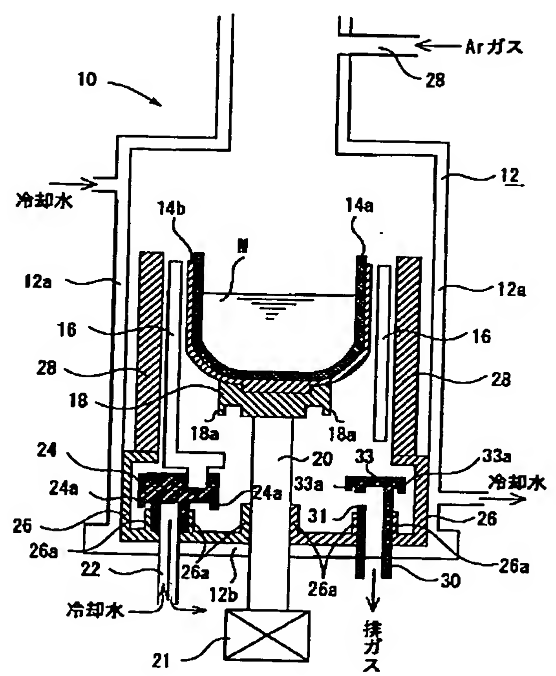
最終頁に続く

(54)【発明の名称】　半導体単結晶製造装置及び黒鉛部材評価方法

(57)【要約】　（修正有）
【課題】　ＣＺ法による半導体単結晶製造装置において不慮の原因により高温の原料が流出した場合に、製造装置外への流出を防止すると共に、流出原料による装置炉壁の浸食を防止する。またそれに適した黒鉛部材の評価方法を提供する。
【解決手段】　製造装置炉１２の内部には、石英製ルツボ１４ａがあり、原料溶融液Mが流出したときに接触の可能性のある保護ルツボ１４ｂ、ルツボ台１８、ルツボ支持軸２０は、黒鉛部材製とした。黒鉛部材として、かさ密度が１．７０～１．９０ｇ／ｃｍ³の等方性黒鉛を用いる。また溶融したシリコン融液に１０分以上２０分以内浸漬し、その後のシリコン融液に浸漬した部分の寸法変化率が５％以下である黒鉛材を使用する。さらに、灰分が２０ｐｐｍ以下の等方性黒鉛を用いる。



【特許請求の範囲】

【請求項１】　製造装置の炉内にルツボに収容された原料融液と原料融液を加熱溶融するための加熱ヒータと製造装置炉壁の保護と炉内の保温等のために配置された黒鉛部材とを有するチョクラルスキー法により単結晶を育成する半導体単結晶の製造装置において、少なくとも原料融液を収容したルツボから溶融原料が流出した際に流出した原料融液と接する可能性のある製造装置炉内に配置された黒鉛部材、あるいは流出した原料融液を保持し製造装置炉壁に接触するのを防ぐための製造装置炉内に配置された黒鉛部材に、かさ密度が１．７０～１．９０ｇ／ｃｍ^{3}の等方性黒鉛を該黒鉛部材の材料として用いることを特徴とする半導体単結晶の製造装置。

【請求項２】　製造装置の炉内にルツボに収容された原料融液と原料融液を加熱溶融するための加熱ヒータと製造装置炉壁の保護と炉内の保温等のために配置された黒鉛部材とを有するチョクラルスキー法により単結晶を育成する半導体単結晶の製造装置において、少なくとも原料融液を収容したルツボから溶融原料が流出した際に流出した原料融液と接する可能性のある製造装置炉内に配置された黒鉛部材、あるいは流出した原料融液を保持し製造装置炉壁に接触するのを防ぐために製造装置炉内に配置された黒鉛部材の材料として、溶融したシリコン融液に１０分以上２０分以内浸漬しその後のシリコン融液に浸漬した部分の寸法変化率が５％以下である黒鉛材を使用することを特徴とする半導体単結晶の製造装置。

【請求項３】　請求項１又は請求項２に記載した半導体単結晶の製造装置であって、少なくとも前記半導体単結晶製造装置の炉内に配置された黒鉛部材の中で、ルツボから原料融液が流出したさいに流出融液と接する可能性のある黒鉛部材が、黒鉛ルツボ、ルツボ台、ルツボ支持軸、黒鉛保護電極部、加熱ヒータ、排ガス管及び又は湯漏れ受け皿であることを特徴とする半導体単結晶の製造装置。

【請求項４】　請求項１～請求項３のいずれか１項に記載した半導体単結晶の製造装置であって、前記黒鉛部材の材料として灰分が２０ｐｐｍ以下の等方性黒鉛を用いることを特徴とする半導体単結晶の製造装置。

【請求項５】　加熱溶融して得た半導体単結晶の原料融液に黒鉛材を浸漬し、原料融液に浸漬した黒鉛材料部分の寸法あるいは体積変化から該黒鉛材の特性を評価することを特徴する半導体単結晶製造装置用黒鉛材の評価方法。

【請求項６】　請求項５に記載した半導体装置用の黒鉛材評価方法であって、評価対象となる黒鉛材を浸漬する前記原料融液は、多結晶シリコンあるいは単結晶シリコンを加熱溶融して得たシリコン融液であることを特徴とする半導体装置用黒鉛材の評価方法。

【発明の詳細な説明】

【０００１】

【発明が属する技術分野】本発明は、半導体単結晶を製造するための半導体単結晶の製造装置とその製造装置に用いる黒鉛部品材料の評価方法に関し、より詳しくは単結晶製造装置炉内のルツボに収容された原料融液に結晶を浸漬し引上げることによって種結晶の下方に単結晶を成長するチョクラルスキー法（Czochralski Method、以下ＣＺ法と称す。）を用いた半導体単結晶の製造装置を構成する黒鉛部品とその黒鉛部品の特性を評価する黒鉛材評価方法に関する。

【０００２】

【従来の技術】従来より半導体単結晶の製造方法の一つとして、半導体製造装置のルツボ内で半導体単結晶原料を融解し、種結晶を該融液面に接融させて半導体単結晶を育成するＣＺ法が広く知られている。ＣＺ法を用いた半導体単結晶の育成技術は、シリコンやＧａＡｓ等の半導体単結晶を得るために幅広く利用されているものであるが、以下ではシリコン半導体単結晶の育成をその一例として本発明の従来例を説明する。

【０００３】ＣＺ法によりシリコン単結晶を育成する方法は、原料となる多結晶シリコンを製造装置炉内に備えられた外側が黒鉛製で内側が石英製で構成されたルツボに充填し、製造装置炉内をアルゴン（以下、Ａｒとも称す。）等の不活性ガスで満たした後に、ルツボの外側周囲に配設された加熱ヒータにより１４００℃以上の高温に加熱して原料を融解する。そして、この加熱溶融されたシリコン融液に上方から種結晶を降ろしシリコン融液面に先端を接融させ、種結晶とシリコン融液の温度が安定したら種結晶を静かに回転させながら上方に引上げることによって、種結晶の下方にシリコンの半導体単結晶を形成するものである。

【０００４】このような半導体単結晶の育成において、単結晶の成長を行っている間は、その原料である溶融液を半導体単結晶の育成が可能な高温の状態にルツボ内で保持し続ける必要がある。この間、地震やその他の不慮の事故によりルツボ内から高温の融液が製造装置炉内に漏れ出した場合には大きな災害を誘発する可能性も十分考えられるため、万が一、ルツボから高温の融液が製造装置炉内に流出したとしても速やかにルツボ内の融液流出を検出し、ルツボから流出した原料融液も製造装置の外で漏れ出さないような対策が施されている。

【０００５】例えば、特開平１１－１８０７９４号公報には、ルツボから原料融液が流出した際にルツボ内での異常な融液面の低下を速やかに検出し、ルツボ内から融液が漏れていることを作業者に知らせる装置が開示されている。そして、ルツボから流出した原料融液に関し特開平２－２２５３９３号公報には、製造装置炉壁の保護や炉内の保温等のために配置された黒鉛部材や断熱材の構成や形状を工夫することにより、ルツボから流出した原料融液を製造装置炉内に保持し製造装置の外側に漏れ出したり、あるいは金属でできた製造装置炉壁を冷却す

るために炉壁を二重構造とし炉壁間に冷却水を還流する等した単結晶製造装置の炉壁を高温の原料融液が侵食するのを防止して、水蒸気爆発等による事故を未然に防ぐための機能を備えた単結晶製造装置が示されてしいる。

【０００６】これらの対策を取ることによって、ルツボから流出した原料融液が製造装置炉外へ漏れて作業者を危険にさらしたり、火災の発生や製造装置を損傷させる等して単結晶の生産が不可能となる事態を回避している。中でも炉内の高温雰囲気から金属製の製造装置炉壁を守るために炉壁に冷却水を還流した単結晶製造装置では、わずかでも高温の融液により炉壁が侵食されると水蒸気爆発等の大きな災害を引き起こす可能性もあり、ルツボから漏れた高温の原料融液は上記のような装置や方法により製造装置炉内に配置された黒鉛部材等で確実に受け止めることが肝要である。

【０００７】

【発明が解決しようとする課題】しかし、単結晶製造装置の炉内部を構成する黒鉛製のルツボや加熱ヒータあるいは高温の輻射熱から炉壁を保護している黒鉛部材の形状や構造を適切なものとし、原料融液がルツボから漏れた場合でもそれを速やかに察知して単結晶製造装置を停止したとしても、製造装置炉内の温度が低下して安全な温度に達すまでには３～５時間にもおよぶ放冷時間が必要であり、その間、漏れた原料融液を製造装置炉外へ流出させたり炉壁に触れさせることなく、確実に製造装置炉内の黒鉛製の部材や断熱材により保持して置かなければならない。

【０００８】特に最近のシリコン半導体単結晶の製造においては、大直径の半導体単結晶を育成するために１００ｋｇ以上もの大容量のシリコン融液が収容可能な大型の単結晶製造装置の導入も進み、このような大型の単結晶製造装置では装置そのものの熱容量も大きく、加熱ヒータへの電力供給を停止して装置内部の温度が下がり安全に開放できるようになるまでには更に長い時間放冷を行う必要がある。当然のことながら、このような状況下でルツボから原料融液が流出した場合であっても、製造装置炉内温度が安全な温度に到達するまでは炉内部を構成する黒鉛部材や黒鉛製断熱材により流出した融液を外部へ漏らすことなく確実に保持しておく必要があり、漏れた融液を確実に保持する為にはより信頼性の高い材料を選択し流出融液と接する部材を作ることが重要となる。

【０００９】また、半導体単結晶の育成にあたっては、単結晶品質の維持と製造装置炉内を構成する部品の保護のために製造装置炉内を反応性の低いＡｒ等の不活性ガスで満たして操業を行っているが、ヒータにより高温に加熱された原料融液からは侵食作用のあるＳｉＯ（シリコンの酸化物）等の物質が常に蒸発しており、これら蒸発物によって製造装置炉内に配置された黒鉛部材や黒鉛製断熱材は侵食を受け、各部材を形成する黒鉛材料の種類によっては強度や保温機能を操業中の侵食により失う可能性がある。ルツボからの流出融液が接する製造装置炉内の黒鉛部材には、原料融液から蒸発した蒸発物に長時間さらされた場合においてもその機能を損なうことなく安定した機能を発揮することも求められる。

【００１０】本発明はこのような問題に鑑みて成されたものであり、ＣＺ法による半導体単結晶製造装置において、不慮の原因により単結晶製造装置炉内部に置かれたルツボから高温の原料融液が流出した場合に、流出原料を炉内に確実に止め製造装置の外へ流出させることなく、更には半導体単結晶製造装置の炉壁も高温の原料融液により浸食されず安全に原料融液を保持する機能を有する製造装置炉内に配置される黒鉛部材を備えた半導体単結晶の製造装置と、それに適した黒鉛部材の材料となる黒鉛材を適切に選択することが可能な半導体単結晶製造装置を構成する黒鉛部材の材料評価方法を提供することを目的とする。

【００１１】

【課題を解決するための手段】上記の課題を解決するために、本発明の半導体単結晶製造装置の第１の態様は、製造装置の炉内にルツボに収容された原料融液と原料融液を加熱溶融するための加熱ヒータと製造装置炉壁の保護と炉内の保温等のために配置された黒鉛部材とを有するチョクラルスキー法により単結晶を育成する半導体単結晶の製造装置において、少なくとも原料融液を収容したルツボから溶融原料が流出した際に流出した原料融液と接する可能性のある製造装置炉内に配置された黒鉛部材あるいは流出した原料融液を保持し製造装置炉壁に接触するのを防ぐための製造装置炉内に配置された黒鉛部材に、かさ密度が１．７０～１．９０ｇ／ｃｍ^{3}の等方性黒鉛を該黒鉛部材の材料として用いることを特徴とする。

【００１２】このように、ルツボから原料融液が漏れた場合に流出融液と接する位置にある黒鉛部材を、原料融液の侵食を受け難い材料を選択して配置しておくことが望ましく、この条件を満たす黒鉛部材の材料として冷間静水圧成形（Cold Isostatic Press、以下ＣＩＰと称する。）を用いて成形製造された、かさ密度が１．７０～１．９０ｇ／ｃｍ^{3}の特性を持つ等方性黒鉛を使用するのが最も好ましい。

【００１３】かさ密度が１．７０ｇ／ｃｍ^{3}以下の黒鉛材を用いて原料融液と接する黒鉛部材を作成した場合には、原料融液が黒鉛部材内部に急速に浸透し急激な体積膨張が生ずるため構造体としての機能を保つことが不可能となり、原料融液を炉内に止めることができず製造装置炉壁等の金属部を侵食させたり、装置炉外へと漏れた融液を流出させる可能性がある。また、例えかさ密度が１．７０ｇ／ｃｍ^{3}以上であったとしても等方性黒鉛以外の押し出し成形（Extruding Press）や型押し成形（Molding Press）等を用いて作られた異方性黒鉛で

は、単結晶育成時に原料融液から出る蒸発物の侵食を受けやすく耐久性や信頼性の面で問題がある。従って、ルツボから原料融液が漏れた場合に流出原料と接する可能性のある黒鉛部品には、かさ密度が１．７０ｇ／ｃｍ^{3}以上の等方性黒鉛を使用するのが適している。しかし、この一方でかさ密度の値が必要以上に高い等方性黒鉛材は製造コストもかさみ非常に高価なものとなるので、かさ密度の上限は１．９０ｇ／ｃｍ^{3}程度に止めておくのが適当である。

【００１４】そして、少なくとも半導体単結晶の炉内に配置された黒鉛部材の中で、ルツボから原料融液が流出したさいに流出融液と接する可能性がある黒鉛ルツボ、ルツボ台、ルツボ支持軸、黒鉛保護電極部、加熱ヒータ、排ガス管及び湯漏れ受け皿を、上記の特性を有するかさ密度が１．７０～１．９０ｇ／ｃｍ^{3}の等方性黒鉛で作れば、ルツボから原料融液が漏れて製造装置炉内の黒鉛部材に接したとしても、部材に変形やヒビ割れを生じないので、これら黒鉛部材で確実に流出した原料融液を保持することができるものである。

【００１５】また、シリコン半導体の単結晶製造装置においては、かさ密度が１．７０～１．９０ｇ／ｃｍ^{3}の等方性黒鉛で炉内黒鉛部材を作り配置すれば、単結晶育成時に原料融液から蒸発するＳｉＯ（シリコンの酸化物）が徐々に黒鉛部材内部に浸透しＳｉＣ化することによって生じるヒビ割れや部材の膨張あるいは強度の低下を抑制することができる。これにより黒鉛部材の耐久性が向上し長時間の使用に耐えることができるようになるとともに、黒鉛部品そのものの信頼性も高まりより確実な融液漏れの対策を講じることが可能となる。

【００１６】本発明のＣＺ法により半導体単結晶を製造する半導体単結晶製造装置の第２の態様は、製造装置の炉内にルツボに収容された原料融液と原料融液を加熱溶融するための加熱ヒータと製造装置炉壁の保護と炉内の保温等のために配置された黒鉛部材とを有するチョクラルスキー法により単結晶を育成する半導体単結晶の製造装置において、少なくとも原料融液を収容したルツボから溶融原料が流出した際に流出した原料融液と接する可能性のある製造装置炉内に配置された黒鉛部材あるいは流出した原料融液を保持し製造装置炉壁に接触するのを防ぐために製造装置炉内に配置された黒鉛部材の材料として、溶融したシリコン融液に１０分以上２０分以内浸漬しその後のシリコン融液に浸漬した部分の寸法変化率が５％以下である黒鉛材を使用することを特徴とする。

【００１７】単結晶製造装置の炉内に配置されたルツボから漏れた高温の原料融液と接する可能性のある黒鉛部材の材料としては、育成する単結晶と同じ原料融液中に黒鉛材を沈めた時に、その浸漬部分の寸法の変化率が５％以下の黒鉛材料を用いても、本発明の第１の態様の半導体単結晶製造装置と同様の特徴を持つ単結晶製造装置を得ることができる。

【００１８】原料融液に黒鉛材を一定時間浸漬させた際の寸法変化率が５％以内の黒鉛材を用いて高温の流出融液と接する可能性のある黒鉛部材を作れば、原料融液と接したときに大きな変形やヒビ割れ、膨張、あるいは強度低下等を起こすことなく黒鉛部材としての特性を維持することができる。また、単結晶育成時に発生するＳｉＯ等の原料融液からの蒸発物にも侵食され難いので、部材の耐久性が高く機能を損なうことなく長時間にわたる使用にも十分耐えることができ、このような材料を用いた黒鉛部材を使用して単結晶製造装置を作れば、不慮の原因によりルツボから原料融液が漏れた場合でも被害を最小限に抑えることが可能な信頼性の高い製造装置とすることができる。

【００１９】そして、黒鉛材を浸漬する融液がシリコンである場合は、融液にテスト部材を浸漬する時間は１０分以上とすべきである。シリコン融液に浸漬する時間が１０分以下では、シリコン融液が十分に材料内部に浸透しないので材料の正しい特性を知ることができないし、また、必要以上に長く浸漬しても得られる結果に大きな違いは見られなくたるため、テスト部材を融液に浸漬しておく時間は長くとも２０分程度以下に止めておくのが効率的である。

【００２０】そして、少なくとも半導体単結晶の炉内に配置された黒鉛部材の中で、ルツボから原料融液が流出したさいに流出融液と接する可能性がある黒鉛ルツボ、ルツボ台、ルツボ支持軸、黒鉛保護電極部、加熱ヒータ、排ガス管及び湯漏れ受け皿を、上記の特性を有する溶融したシリコン融液に１０分以上２０分以内浸漬しその後のシリコン融液に浸漬した部分の寸法変化率が５％以下である黒鉛材で作れば、ルツボから原料融液が漏れて製造装置炉内の黒鉛部材に接したとしても、部材に変形やヒビ割れを生じないので、これら黒鉛部材で確実に流出した原料融液を保持することができるものである。

【００２１】また、半導体単結晶の製造装置に用いられる黒鉛部材の純度は、育成結晶品質への不純物の影響を考慮すれば高純度であることが望ましく、流出融液と接する可能性のあるこれら黒鉛部材も「ＪＩＳ規格　Ｒ７２２３」で測定した時の灰分が２０ｐｐｍ以下である黒鉛材を用いて構成すればより半導体単結晶製造に適した装置とすることができる。

【００２２】単結晶製造装置用黒鉛材の特性を知るための本発明による半導体単結晶製造装置用黒鉛材の評価方法は、加熱溶融して得た半導体単結晶の原料融液に黒鉛材を浸漬し、原料融液に浸漬した黒鉛材料部分の寸法あるいは体積変化から該黒鉛材の特性を評価することを特徴とする。

【００２３】半導体単結晶の製造装置内部を構成する黒鉛部材の特性を評価するにあたり、炉内部材として使用する黒鉛材からテスト試料を切り出して製造装置内で溶融された単結晶の原料融液にテスト試料を浸漬し、テス

ト試料の浸漬部分の形状や体積が浸漬前後でどのように変化したか寸法あるいは体積を比較すれば、製造装置炉内を構成する部材として適切なものであるか、また、製造装置炉内のどの部分の部材として使用可能であるかを適切に評価することがきる。特に、原料融液と接する可能性のある黒鉛材料の特性を評価する場合には的確に原料付着時の侵食度合いを知ることができるので、より適切に求める特性を有する黒鉛材料を選択することが可能となる。

【００２４】また、これら黒鉛材から切り出したテスト試料を浸漬する融液には、多結晶シリコンあるいはシリコン単結晶を溶融してできた融液を用いてもよい。黒鉛は溶融されたシリコン融液に侵食されやすいので反応が速く進み、黒鉛のテスト試料をシリコン融液に浸漬してその特性を評価すれば、比較的短い時間で評価結果を得ることができる。

【００２５】

【発明の実施の形態】以下に本発明の実施の形態を添付図面を参照しながら、ＣＺ法を用いたシリコン単結晶の育成例を挙げて説明するが、本発明はこれらにのみ限定されるものではない。例えば、本発明の半導体単結晶の製造装置やその装置に用いる黒鉛部材の評価方法は、原料融液に磁場を印加しながら半導体単結晶を育成するＭＣＺ法を用いた半導体単結晶の製造装置でも利用することは当然可能であり、更には化合物半導体等の他のＣＺ法を利用した半導体単結晶の製造装置に用いることも勿論可能である。

【００２６】図１は、本発明の単結晶製造装置の一つの実施の形態を示す概略断面図である。図１に示す単結晶製造装置１０の製造装置炉１２は、石英製ルツボ１４ａに収容された原料融液Ｍを溶融する加熱ヒータ１６等からの高温の輻射熱による温度上昇を抑制し、常時一定温度とするため製造装置炉壁１２ａを二重構造とし二重の壁の間に冷却媒体を還流して製造装置炉壁１２ａが高温とならないよう保護されている。本発明の製造装置１０では冷却媒体として水を還流し使用している。

【００２７】また、製造装置炉１２の内部中央には原料融液Ｍを収容した石英製ルツボ１４ａが配置されており、この石英製ルツボ１４ａは石英製であることから衝撃に弱く、さらには１４００℃以上もの高温に加熱された場合には石英製ルツボ自体も軟化することから、それを保護し支えるために外側に黒鉛から作られた黒鉛製ルツボ１４ｂを配している。そして、この黒鉛製ルツボ１４ｂはルツボ台１８を介してルツボ支持軸２０によって支持されており、ルツボ支持軸２０の下端に取付けられたルツボ回転駆動機構２１によって回転自在及び上下動自在とされている。

【００２８】ルツボ台１８とルツボ支持軸２０も黒鉛材で作られており、石英製ルツボ１２ａを保護するための黒鉛製ルツボ１２ｂも併せて、石英製ルツボ１２ａから高温の原料融液Ｍが流出した場合には原料融液Ｍと接する可能性が一番高いと考えられるため、かさ密度１．７０～１．９０ｇ／ｃｍ³の等方性黒鉛材を使用している。

【００２９】また、ルツボ台１８の下側面外周には石英製ルツボ１４ａから漏れた原料融液Ｍがルツボ支持軸２０に回り込むのを防止するため、下に向かって凸となる突条１８ａが設けられている。石英製ルツボ１４ａから原料融液Ｍが漏れ出してルツボ台１８に達した時に、この突条１８ａによって漏洩融液がルツボ支持軸２０に回り込むのを可能な限り防ぎ、ルツボ支持軸２０を伝って炉外へ漏れ出すことを防止している。

【００３０】一方、黒鉛ルツボ１４ｂの直ぐ外側には原料融液Ｍを加熱溶融するための黒鉛製の加熱ヒータ１６が置かれており、この加熱ヒータ１６はヒータを加熱する電流を供給する金属製の電極部２２によって支えられている。金属製電極部２２は、製造装置炉１２内の高温雰囲気から保護することと、石英製ルツボ１２ａからの原料融液Ｍの流出があった場合に漏洩融液による侵食を受けないように黒鉛保護電極部２４を介して加熱ヒータ１６と接合され、加熱ヒータ１６への電力供給を行っている。

【００３１】なお、金属製電極部２２も必要以上に加熱するのを防止するため、製造装置炉壁と同様に冷却水が還流されている。また、黒鉛保護電極部２４の周縁部下面にも原料融液Ｍがルツボ１４ａから漏洩した場合に電極等を伝って炉外へ漏れることのないよう、下方に向かって凸となる突条２４ａが廻らされており、この突条２４ａによって上方から下流してきた原料融液Ｍを適切に製造装置炉１２の下部に設置された湯漏れ受け皿２６に誘導することができる。そして、加熱ヒータ１６、黒鉛保護電極部２４も石英製ルツボ１４ａから原料融液Ｍが漏れた場合には融液が着液する可能性が非常に高いため、かさ密度１．７０～１．９０ｇ／ｃｍ³の等方性黒鉛材を使用している。

【００３２】更に、加熱ヒータ１６の外周には黒鉛製の断熱材２８を装備し、製造装置炉１２内の保温とヒータ１６等からの輻射熱が直接炉壁１２ａにあたらないように保護している。

【００３３】そして、これら製造装置炉１２内を構成する炉内部材の下方、製造装置炉１２の一番下側には湯漏れ受け皿２６が配置され製造装置炉１２の壁底１２ｂを保護するとともに、不慮の原因によりルツボ１４ａから原料融液が流出した場合には、この湯漏れ受け皿２６に流出融液Ｍを集め受け止めることで高温の原料融液Ｍが金属製の炉壁１２ａを侵食したり製造装置炉１２外へ漏れ出すのを防ぎ、炉内の温度が安全温度まで下がるまでの間、原料融液を保持する役目を担っている。このような理由から、本発明の装置でも湯漏れ受け皿２６の材料として、かさ密度１．７０～１．９０ｇ／ｃｍ³の等方

性黒鉛材を用いている。

【００３４】この湯漏れ受け皿２６は製造装置炉１２の底壁１２ｂを保護する他に、底壁１２ｂに設けられた炉内部材、例えば金属製電極２２、黒鉛製電極部２４、ルツボ支持軸２０及び排ガス管３０を保護する必要があり、それら部材と当接する湯漏れ受け皿２６の部分はそれらの部分を包囲するように環状の立上り部分２６ａとなっている。

【００３５】また、半導体単結晶の育成においては製造装置炉１２内を不活性ガスで満たして操業を行うため、製造装置１０には不活性ガスを炉内に導入するガス導入管２８と、そのガスを排気するために製造装置炉１２内の下部には湯漏れ受け皿２６を貫通するように排ガス管３０も設けられている。本発明の装置では、操業時は製造装置炉の上方にあるガス導入管２８からＡｒガスを導入し原料融液に向けて下流するようにガスを炉内に流し、製造装置炉下部に設けられた排ガス管３０から炉内のガスを外に排出する構造とした。

【００３６】この排ガス管３０も原料融液Ｍが漏れた場合には流出融液と接する可能性があるため上記の炉内黒鉛部材と同様に、かさ密度１．７０～１．９０ｇ／ｃｍ^{3}の等方性黒鉛材で作るか、排ガス管３０が金属である場合は金属部を保護するようにこれらを黒鉛材で保護するのがよい。本発明の単結晶製造装置１０では排ガス管３０を上記特性の等方性黒鉛材で作り、漏れた原料融液Ｍが湯漏れ受け皿２６まで達した時に排ガス管３０の排ガス入口３１から流出しないよう排ガス管３０を湯漏れ受け皿２６の底部から立設させて排ガス入口３１を上方に位置させ、更には排ガス管３０の上方から落ちてくる原料融液Ｍを受けるために排ガス管３０の上端には上端壁３３が形成されている。この上端壁３３の周縁部下面には排ガス入口３１から漏洩融液が外に漏れることのないように、下方に向って凸となる突条３３ａが突設されており、上端壁３３に落下した融液はこの突条３３ａを伝わって、湯漏れ受け皿２６に集められる。

【００３７】なお、本発明において黒鉛材のかさ密度とは、黒鉛材の単位体積当たりの質量（ｇ／ｃｍ^3）のことをいう。そして、これら黒鉛部材の材料として寸法変化率が５％以下の黒鉛も使用可能である。

【００３８】上記した本発明の半導体単結晶製造装置を用いて単結晶を育成するには、石英製ルツボ１４ａ内に多結晶シリコン原料を仕込み、炉内を不活性ガスで満たした後に加熱ヒータ１６でシリコンの塊である多結晶シリコン原料を溶かし融液とすることで単結晶を育成する原料融液Ｍを得る。

【００３９】その後、上方から単結晶を引上げるためのワイヤ（不図示）を降ろして、ワイヤ下端に取り付けられた種結晶（不図示）を原料融液Ｍに浸漬した後、温度が安定したところで静かに引上げワイヤを巻き取ることによって、種結晶の下方に半導体単結晶を育成するものである。

【００４０】また、本発明の黒鉛部材の材料となる黒鉛材の原料融液に対する侵食性の評価は、部材となる黒鉛材から１辺Ｘが２０ｍｍ四方で長さＹが１００ｍｍの図２に示す棒状の浸漬テスト試料Ｔを作製し、種結晶に替えてこの浸漬テスト試料を製造装置炉１２内の引上げワイヤー３５の先端に取り付け、装置炉１２内を単結晶育成時と略同等の条件とした後に、浸漬テスト試料Ｔの先端が２０～３０ｍｍ程度原料融液に沈むまで静かに降下させテスト試料Ｔの浸漬を行った。なお、図２において、図１と同一又は類似部材は同一符号で示されている。

【００４１】テスト試料Ｔが所定量原料融液に沈んだ後、所定時間そのままの状態でテスト試料Ｔを保持しその後融液Ｍから引上げて、原料融液Ｍに浸漬する前後での同方向の寸法の変化率（Δｄ）を測定し、原料融液Ｍに対するテスト試料の耐侵食性の評価を行うものである。

【００４２】なお、寸法変化率（Δｄ）は下記式（１）によって計算する。

【００４３】

【数１】

$$\Delta d = \frac{X_2 - X_1}{X_1} \times 100(\%) \quad \cdots \quad (1)$$

【００４４】式（１）においてX_1はシリコン融液に浸漬前のテスト試料Ｔの幅及びX_2はシリコン融液に浸漬後のテスト試料Ｔの幅である。

【００４５】

【実施例】（実施例１）かさ密度が、１．７４、１．７７、１．８５ｇ／ｃｍ^{3}の等方性黒鉛材から１辺が２０ｍｍ四方で長さ１００ｍｍの浸漬テスト試料を作製した。

【００４６】これら浸漬テスト試料を図２に示したような製造装置の引上げワイヤー先端に取り付け、製造装置炉内のルツボ上方に配置し、その後製造装置炉内の石英製ルツボに１０ｋｇの多結晶シリコンを充填した後にヒータを加熱して溶解し、融液温度が１４２０℃前後のシリコンの原料融液を得た。

【００４７】融液温度が安定したところで引上げワイヤーを巻き出して浸漬テスト試料を融液面直上まで降ろし３０分間加温した後に、シリコン融液にテスト試料先端を長さ方向に２０ｍｍ浸漬した。シリコン融液内に１０分間テスト試料を止め、その後引上げワイヤーを静かに巻き上げてテスト試料を融液から取り出した後に試料温度を常温まで徐々に戻し浸漬部分の寸法の変化測定と目視による外観観察を行い黒鉛材の特性を評価した。

【００４８】この評価では表１と図３に示されるように、１．７０ｇ／ｃｍ^{3}の等方性黒鉛材では寸法変化率が４％以下に止まり、極端な膨張やヒビ割れは観察され

ず原料融液に浸漬する前と略同じ形状を保っていた。

（比較例１）同様に、かさ密度が１．６５、１．６８ｇ／ｃｍ^{3}の押出し成形による異方性黒鉛材と、かさ密度が１．６８ｇ／ｃｍ^{3}の型押し成形による異方性黒鉛材から実施例１を同様の形状の浸漬テスト試料を作製し、実施例１と同一条件下でシリコン融液への浸漬を行った。＊

＊【００４９】結果は表１及び図４に示す通りであり、全ての試料において浸漬部分の寸法変化率は５％を超え、浸漬部分にヒビ割れが生じたものや形状が大きく変化（膨張）したものがほとんであった。

【００５０】

【表１】

（　黒鉛材のシリコン融液への浸漬評価結果）

実施例 比較例	黒鉛材品種	成形方法	かさ密度 （g／cm^3）	融液浸漬後の 寸法変形率	外観観察結果 （ヒビ割れ、変形等）
実施例１	等方性黒鉛	冷間静水圧成形	１．７４	１～２％	亀裂、変形等なし
		冷間静水圧成形	１．７７	１～２％	亀裂、変形等なし
		冷間静水圧成形	１．８５	１％以下	亀裂、変形等なし
比較例１	異方性黒鉛	押出し成形	１．６５	５％以上	浸漬部分が大きく膨張 亀裂が数多く発生
		押出し成形	１．６８	５％以上	浸漬部分が大きく膨張 亀裂が数多く発生
		型押し成形	１．６８	５％以上	浸漬部分が大きく膨張 亀裂が数多く発生

【００５１】（実施例２）次に、製造装置炉内に配置する黒鉛部材の中で、原料融液がルツボから漏れた際に原料融液と接する可能性のある、黒鉛製ルツボ、ルツボ台、ルツボ支持軸、加熱ヒータ、黒鉛保護電極部、排ガス管、湯漏れ受け皿をかさ密度が１．７４～１．７７ｇ／ｃｍ^3、灰分が２０ｐｐｍ以下の高純度の等方性黒鉛材で作りシリコン半導体単結晶の製造に使用した。

【００５２】そして、半導体単結晶の育成を行うにあたり口径６０ｃｍの石英製ルツボに多結晶シリコン原料を１５０ｋｇ仕込み溶融を開始したところ、約８０％の原料が融液となったところで石英製ルツボに亀裂が入り、ルツボから融液が漏れ出していることがわかった。直ちに加熱ヒータの電源を切り、装置冷却のため炉壁還流している冷却水の温度を確認したところ、水温は１．２℃程上昇していたが特に異常は見られなかった。

【００５３】製造装置内部の温度が常温近くまで低下した後に、製造装置を解体して原料融液の流出状況を確認したところ、製造装置の底部に置かれている湯漏れ受け皿にはおよそ８０ｋｇ前後のシリコンが凝固していたが、流出した原料が更に湯漏れ受け皿から漏れた形跡はなく炉壁は完全に保護されていた。また、ルツボからの流出原料が接したと思われる、黒鉛製ルツボ、ルツボ台、湯漏れ受け皿を観察したところ原料融液によって侵食を受けた様子は観察されず予定した通りの機能を発揮できていることがわかった。

【００５４】なお、本発明は上記した実施の形態に限定されるものではない。上記した実施の形態は単なる例示であり、本発明の特許請求の範囲に記載された技術的思想と実質的に同一な構成を有し、同様の効果を奏するものであればいかなるものであっても、本発明の技術的範囲に包含されることは無論である。

【００５５】例えば、本発明の半導体単結晶の製造装置を原料融液に磁場を印加しないで単結晶を育成するＣＺ法を例に挙げて説明したが、原料融液に磁場を印加しながら半導体単結晶を育成するＭＣＺ法を用いた半導体単結晶の製造装置においても同様の効果が得られることは言うまでもない。そして、本発明の半導体単結晶製造装置や半導体単結晶製造装置に用いられる黒鉛部材の特性を評価する方法も、シリコン以外の半導体単結晶の製造装置やその装置を構成する黒鉛部材の特性を評価するのに利用可能なことは当然であり、ＣＺ法を用いた例えばＧａＡｓ結晶等の化合物半導体の育成に適用した場合でもその効果を十分に発揮することができる。

【００５６】

【発明の効果】以上に述べたごとく、ＣＺ法による半導体単結晶の製造装置を本発明の装置の構造とすれば、製造装置炉内を構成する黒鉛部材に侵食性のある原料融液が接したとしても黒鉛部材に大きな変形やヒビ割れを生じることが無いので、例え不慮の原因により装置炉内に配置されたルツボから高温の原料融液が漏れたとしても、漏れた原料融液を炉内に配置された湯漏れ受け皿等の流出融液を保持するための黒鉛部材へ正確に導くことができるとともに、ルツボ内からの原料融液の漏れを確認してから製造装置を停止し炉内の温度が安全な温度に低下するまでの長時間にわたり、これら黒鉛部材により流出融液を確実に炉内に止めておくことが可能となる。

【００５７】これによってルツボから漏れた原料融液が製造装置の外へ流れ出ることが無くなるため、流出した高温の融液により装置を破損することが回避可能となるばかりか、装置近くで働いている作業者を危険にさらすことなく安全にトラブルに対処できるようになる。

【００５８】更には、ルツボから流れ出た侵食性の高い高温の原料融液が、冷却水を還流した金属製の製造装置炉壁やヒータ電極、ルツボ駆動軸等に直接接することも

無くなるので、高温の液体が水等の冷却媒体と混じり合うことによって起こる水蒸気爆発等の重大な災害も回避することができる。

【００５９】そして、本発明の評価方法を用いて単結晶製造装置炉内の黒鉛部材として使用する黒鉛材料を評価すれば、それぞれの単結晶製造装置に適した黒鉛材料を簡単に、そしてより確実に知ることができるようになり、これら製造装置内に配置する黒鉛部材の信頼性も一層向上するものである。

【図面の簡単な説明】

【図１】　本発明の単結晶製造装置の一つの実施の形態を示す概略断面説明図である。

【図２】　実施例１の浸漬テストに用いられた製造装置の概略断面説明図である。　＊

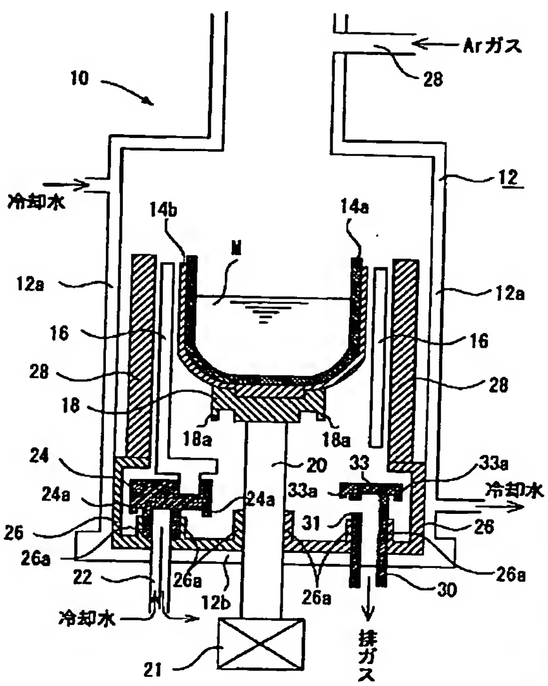
＊【図３】　実施例１における浸漬後の浸漬テスト試料の表面状態を示す写真である。

【図４】　比較例１における浸漬後の浸漬テスト試料の表面状態を示す写真である。

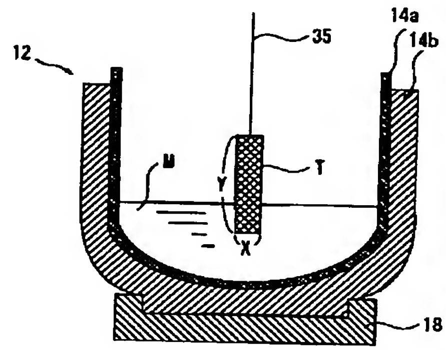
【符号の説明】

１０：製造装置炉、１４ａ：石英製ルツボ、１４ｂ：黒鉛製ルツボ、１６：加熱ヒータ、１８：ルツボ台、１８ａ：突条、２０：ルツボ支持軸、２１：ルツボ回転駆動機構、２２：金属製電極部、２４：黒鉛保護電極部、２４ａ：突条、２６：湯漏れ受け皿、２６ａ：立上り部分、２８：断熱材、３０：排ガス管、３１：排ガス入口、３３：上端壁、３３ａ：突条、３５：引上げワイヤー、Ｍ：原料融液、Ｔ：浸漬テスト試料。

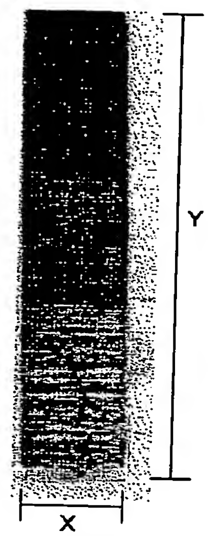
【図１】



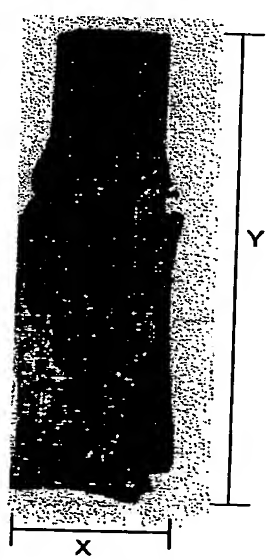
【図２】



【図３】



【図４】



フロントページの続き

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